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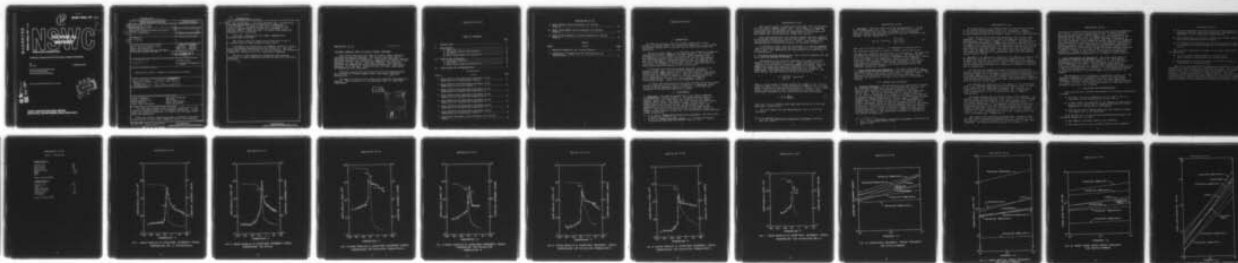
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TORSIONAL PENDULUM STUDY ON SEVERAL RUBBER COMPOUNDS.(U)  
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## TECHNICAL REPORT

WHITE OAK LABORATORY

TORSIONAL PENDULUM STUDY ON SEVERAL RUBBER COMPOUNDS

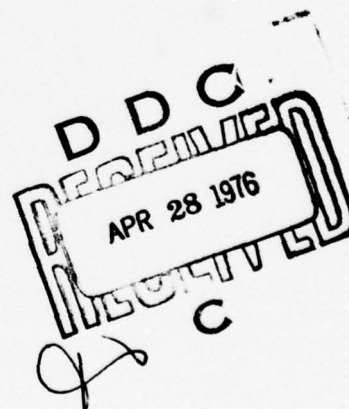
BY  
G.F. Lee

4 JANUARY 1977

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) <p>The Navy has frequent need of acoustic properties in the kilohertz frequency region of viscoelastic materials. In this study the acoustic properties of interest are the shear sound speed and absorption.</p> <p>→ The goal of this investigation was to determine the shear properties on various rubber compounds using a low frequency →</p>		

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① 1 Hz) measurement - torsional pendulum (TP) - to determine the damping and modulus as a function of temperature (-175 to 800 C). By applying WLF shift the damping and modulus were presented as a function of frequency (1 to 100 kHz, 100 C). From this data, the shear sound speed and absorption were calculated.

From this investigation the rubber compounds are characterized as follows:

The glass transition temperatures were found to be in a relative narrow range between -45 and -330 C.

By changing the formulation the damping varied by a factor of 6; the modulus changed as much as a factor of a 330; the sound speed varied by a factor of 16; and finally, the absorption changed by a factor of 10, as a function of frequency.

The TP is a good method for determining the acoustic properties of rubber compounds provided the WLF shift constants are known.

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#### TORSIONAL PENDULUM STUDY OF SEVERAL RUBBER COMPOUNDS

This report describes work done on several rubber compounds intended for acoustic purposes. The goal of this work was to determine the mechanical properties - logarithmic decrement (damping) and shear modulus - as a function of temperature by utilizing a low frequency ( $\sim 1$  Hz) method - torsional pendulum. Then applying WLF shift the damping and modulus were presented as a function of frequency (1 to 100 kHz). From this data, the shear sound speed and absorption were calculated.

This work was carried out during FY 1976 with funds provided by the Naval Air Systems Command under Task Number A320-5203/WF00-543-201.

The materials used in this report were obtained from commercial sources. Their evaluation by the Laboratory does not imply Navy endorsement.

*J R Dixon*  
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## I INTRODUCTION

The Navy has frequent need of acoustic properties in the kilohertz frequency region for viscoelastic materials. In this study the acoustic properties of interest are the shear sound speed and absorption.

The goal of this report is to determine the mechanical properties logarithmic decrement (damping) and shear modulus - as a function of temperature (-175 to 80°C) on various rubber compounds with approximately the same glass transition ( $T_g$ ) using a low frequency ( $\sim 1$  Hz) measurement - torsional pendulum (TP). By applying the WLF' shift the damping and modulus were shifted and presented as a function of frequency (1 to 100 kHz, 100°C). From this data, the shear sound speed and absorption can be calculated.

In the past the torsional pendulum has been utilized in the study of rigid, linear and crosslinked polymers (polymethyl methacrylate (PMMA) epoxies, etc.), but the TP has not been extensively used for compounded rubbers. Therefore, another purpose of this investigation was to extend the testing capabilities of the TP by comparing the results of a linear polymer - PMMA and crosslinked polymer - epoxy with the compounded rubbers.

In brief, the conclusion of this study was that the TP was proven useful in determining the acoustic properties of compounded rubber in the kilohertz frequency region of interest, if the empirical constants of the WLF shift equation are known.

## II EXPERIMENTAL

a. Materials - The physical properties of the rubber compounds investigated are listed in Table 1. The polybutadiene rubber, a research sample, was supplied by the Firestone Tire and Rubber Company, Akron, Ohio. The rubber was made through the alkali metal polymerization process, providing a rubber of principally 1, 2 configuration with a vinyl content of approximately 62%. The compounding and vulcanization are similar to that reported by Barron.<sup>1</sup> The ratio of the viscosity average molecular weight to number average molecular weight was 2.00, indicating a broad molecular weight distribution.

1. J. D. Ferry, Viscoelastic Properties of Polymers, John Wiley Sons, Incorporated (1970) p. 314.
2. H. Barron, Modern Synthetic Rubbers, D. Van Nostrand Company, Incorporated, New York 2nd edition (1943) p. 174.



The nitrile rubber is commercially available from the Firestone Tire and Rubber Company, Akron, Ohio. This rubber is a copolymer of acrylonitrile and butadiene with a nitrile content of 32%. The compounding and vulcanization are proprietary to the manufacturer. This rubber has a broad molecular weight distribution.

Four polysulfides were also research samples and were supplied by the Thiokol Corporation, Trenton, New Jersey. The four compounds are designated as formulation A, B, C, and D. The components in compounding and vulcanization process are presented in Table 2.

A polysulfide rubber from the 3M Company is a sealing compound made by blending carbon black and other additives to a polysulfide rubber stock. The compounding and vulcanization are proprietary to the manufacturer.

b. Torsional Pendulum Measurements - The torsional pendulum (TP) was built from the designs of Nielsen.<sup>2</sup>

The purpose of the torsional pendulum is to excite the test sample in free oscillation at  $\sim 1$  Hz. These oscillations are recorded on a strip-chart recorder as damped sine waves. From the damped sine waves two mechanical properties are obtained, shear modulus and logarithmic decrement determined as a function of temperature. The temperature range was from  $-175$  to  $80^{\circ}$  C. The shear modulus (G) was calculated using the expression:

$$G = \frac{2.35 LI}{CD^3 \mu P^2} \quad (\text{dynes/cm}^2) \quad (1)$$

where L is the length of sample between the clamps in cm, C is the width of sample in cm, D is the thickness of sample in cm, I is the moment of inertia of the oscillating system in g-cm<sup>2</sup>,  $\mu$  is the shape factor, and P is the period of oscillations in seconds. The logarithmic decrement ( $\Delta$ ) was calculated from the relationship

$$\Delta = \frac{1}{n} \ln \frac{A(r)}{A(r+n)} \quad (2)$$

where A(r) is the reference peak amplitude and A(r+n) is the peak amplitude n cycles later.

The test sample size was approximately  $7.87 \times 1.02 \times 0.23$  centimeter.

3. L. E. Nielsen, Mechanical Properties of Polymers, Reinhold New York (1962).

c. WLF Shift - The data from the TP was determined at  $\sim 1$  Hz and over a broad temperature range. By applying WLF shift to the TP data, the temperature scale may be changed to a corresponding frequency scale. The frequency region of interest is between 1 and 100 kHz. The WLF expression<sup>3</sup> is

$$\log a_T = \log f/f_0 = \frac{-C_1^1 (T-T_1)}{(C_2^1 + T-T_1)} \quad (3)$$

where  $a_T$  is the shift factor,  $T$  is temperature in Kelvin,  $T_1$  is the reference temperature in Kelvin ( $283^\circ\text{K}$ ), and  $C_1^1$  and  $C_2^1$  are the empirical constants characteristic of the material. The empirical constants were obtained from the literature.<sup>4</sup> The shift factor  $a_T$  is the ratio of  $f$  to  $f_0$ , where  $f$  represents the frequency in Hertz, that corresponds to the temperature  $T$  and  $f_0$  is the reference frequency. The reference frequency was determined to be 0.25 Hz. This reference frequency is the average frequency of the rubber compounds in free oscillation in the region between  $T_g$  and the reference temperature ( $283^\circ\text{K}$ ).

d. Shear Sound Speed and Absorption - The shear sound speed was calculated from the shear modulus by using the expression  $v_s = \sqrt{G/\rho}$ , where  $v_s$  is shear sound speed (cm/sec) and  $\rho$  is density (g/cm<sup>3</sup>). The absorption was determined from the logarithmic decrement  $\Delta$  by applying the expression  $\alpha = 8.686 \Delta \pi f/v_s$ , where  $\alpha$  is absorption (dB/cm).

### III RESULTS AND DISCUSSION

a. Torsional Pendulum - The shear modulus and logarithmic decrement as a function of temperature for the polybutadiene, nitrile, and the five polysulfides, are presented in Figure 1 through 7, respectively. The glass transition temperatures ( $T_g$ ) range from  $-45$  to  $-330^\circ\text{C}$ . The  $T_g$  for each material is listed in Table 1. In all cases the magnitude of the damping at temperatures above  $T_g$  was greater than at temperatures below  $T_g$ . For most linear polymers a symmetrical damping peak occurs. The increase in damping is probably due to the effects of crosslinks. The mechanism for a lightly crosslinked rubber to damp out an applied stress, is to convert the energy associated with the applied stress to heat energy by friction. The friction is generated between the oscillating, uncrosslinked chains trying to follow the applied stress, but the crosslinked chains, limited in mobility, impede the more mobile, uncrosslinked chains. The logarithmic decrement is a measure of this effect as shown here.

4. J. D. Ferry, Viscoelastic Properties of Polymers, John Wiley and Sons, Incorporated (1970) p. 303.
5. Ibid, p. 316.

From the glassy to the rubbery state the shear modulus changes on the average of two decades ( $10^9$  to  $10^7$  dynes/cm<sup>2</sup>). Except in the case of polysulfide formulation A, in which the change in the shear modulus spans three decades, Figure 3 ( $10^9$  to  $10^6$  dynes/cm<sup>2</sup>).

Since the TP has not been used extensively in testing rubber compounds, a comparison of the results of PMMA and an epoxy with polybutadiene, as a typical example, will be made. The magnitude of the logarithmic decrement for PMMA and an epoxy is between 0.01 to 3.0 Neper, while the shear modulus ranged from  $10^8$  to  $10^{10}$  dynes/cm<sup>2</sup>. In comparison to polybutadiene, the logarithmic decrement was in the same range but the shear modulus was about one order of magnitude lower than the PMMA and epoxy. From this result the magnitudes of the mechanical properties of polybutadiene are within the testing capabilities of the TP.

b. WLF Shift - By applying the WLF shift the temperature scale on the TP plots was converted to a corresponding frequency scale. The frequency of interest was between 1 and 100 kHz, which corresponds to the region just above  $T_g$  in all cases. The logarithmic decrement and shear modulus are plotted as a function of frequency in Figure 8 and 9, respectively.

In each case the damping increased linearly with frequency. Overall, polysulfide formulation C has the highest damping over the frequency range (1.8 to 6.0 Neper) while polysulfide formulation A has the lowest damping over the same frequencies (0.81 to 0.95 Neper). Polysulfide formulation B and C, nitrile, and polybutadiene appeared more dependent on frequency (steep slope) than polysulfide formulation A and D, and 3M (slight slope).

By changing the formulation of the rubbers in the polysulfide series, the damping properties were varied almost 1 order of magnitude, Figure 8. The logarithmic decrement, for instance, at a frequency of  $10^4$  Hz, increased as follows: .88 - formulation A, 1.44 - formulation D, 2.75 - formulation B, and 3.3 Neper - formulation C.

The shear modulus also increased linearly with frequency. The largest values of shear modulus ( $1.05 \times 10^9$  to  $2.9 \times 10^9$  dynes/cm<sup>2</sup>) were by polysulfide formulation C over the frequency range. While the lowest values ( $8.1 \times 10^6$  to  $8.4 \times 10^6$  dynes/cm<sup>2</sup>) were by polysulfide formulation A. By changing the formulation of rubber in the polysulfide series the shear modulus changes approximately 2 orders of magnitude, Figure 9. The shear modulus, for instance, at a frequency of  $10^4$  Hz, increased as follows:  $8.3 \times 10^6$  - formulation A,  $7.3 \times 10^7$  - formulation B,  $7.4 \times 10^8$  - formulation D, and  $1.79 \times 10^9$  dynes/cm<sup>2</sup> - formulation C.

The accuracy of the mechanical properties - frequency plots - for some rubber compounds are dependent on the constants of the WLF equation, since the constants are characteristic of the rubber compounds. For polysulfide formulation C at a given  $T - T_1$



(equation 3) a greater shift to the left of the reference temperature ( $T_1 = 10^\circ \text{C}$ ) or to lower temperatures (Figure 5) occurs than for polysulfide formulation A at the same  $T - T_1$ . This difference in the magnitude of shift, which is measured by the difference of the shift factor  $a_T$ , between the two rubber compounds is dependent on the shift constants. This dependence becomes important for compounds like polysulfide formulation C but not for polysulfide formulation A, because a shift to lower temperatures for polysulfide formulation C means approaching closer to  $T_g$  than for polysulfide formulation A. By approaching  $T_g$  the mechanical properties change rapidly, therefore a greater variation in the slope of the curves in the mechanical properties - frequency plots occurs in polysulfide formulation C than for polysulfide formulation A for a change in shift constant.

c. Shear Sound Speed and Absorption - The shear sound speed is plotted as a function of frequency in Figure 10. Polysulfide formulation A has the lowest sound speed,  $v_s = 2.26 \times 10^3 \text{ cm/sec}$  and polysulfide formulation C has the highest sound speed,  $v_s = 2.55 \times 10^4 \text{ cm/sec}$ , at  $10^3 \text{ Hz}$ . Polysulfide formulation A was relatively less dependent on frequency than polysulfide formulation B, C and D, 3M, polybutadiene, and nitrile. In the polysulfide series, by changing formulation, the sound speed can be varied over one decade.

The absorption versus frequency is presented in Figure 11. The absorption dependence on frequency was similar for each rubber compound. Polysulfide formulation D has the lowest absorption 1.74 dB/cm, and polysulfide formulation A has the highest absorption 9.9 dB/cm at  $10^3 \text{ Hz}$ . By changing the formulation, the shear sound absorption was varied over 1 decade.

#### IV CONCLUSIONS AND RECOMMENDATIONS

From the torsional pendulum measurements the following conclusions are made:

- The glass transition temperatures were found to lie in a relatively narrow range between  $-45$  and  $-33^\circ \text{C}$ .
- In each rubber the magnitude of the damping at temperatures above  $T_g$  was greater than at temperatures below  $T_g$ . This difference is attributed to crosslinking.
- From the glassy to rubbery state the shear modulus changes two decades ( $10^9$  to  $10^7 \text{ dynes/cm}^2$ )

From the WLF shift of the torsional pendulum measurements the following conclusions are made:

- The damping increased linearly with frequency.
- The shear modulus also increased linearly with frequency.



- The large dependence of shear modulus and damping factor on frequency obviously means large dependence on temperature, especially, in the temperature region near  $T_g$ .
- The polysulfides as a class of rubbers have higher damping than either polybutadiene and nitrile.
- By changing the formulation one can control both the damping by a factor of 6 and shear modulus by as much as a factor of a 330.

The following conclusions are made on the shear sound speed and absorption:

- The shear sound speed changed by a factor of 16 with a change in formulation of the polysulfides.
- The absorption varied by a factor of 10 with a change in formulation of the polysulfides.

The TP is a good method for determining the properties of rubber compounds provided the WLF shift constants are known. The shifted data at best is an approximation since the constants were obtained from the literature. Therefore it may be possible that these constants are not characteristic of these rubbers. It would be useful to determine the damping and shear modulus as a function of frequency in the frequency range of interest, possibly by an acoustic method. Then this data could be compared with the TP data shifted by WLF.

Table 1  
Physical Properties For Various Rubbers

	Density (g/cm <sup>3</sup> )	Shore A Hardness	T <sub>g</sub> (°C) <sup>1</sup>	T <sub>g</sub> (°C) <sup>2</sup>
Polybutadiene (Firestone)	1.115	63	-48	-33
Nitrile (Firestone)	1.123	63	-48	-24
Polysulfide Formulation A	1.585	8		-45
(Thiokol) B	1.666	52		-45
C	1.546	75		-38
D	1.726	73		-42
Polysulfide (3M Co.)	1.387	53		-38

1. Determined by Differential Thermal Analysis (DTA)
2. Determined by Torsional Pendulum (TP)

Table 2

## Components in Compounding and Vulcanization for Polysulfides

FORMULATION A:

<u>PART A</u>	<u>Pbw</u>
Santicizer 160	67
OMYA BLR-3	40
Titanox RA-50	45
Sterling MT	6
Epon 828	1
3 A Linde Sieve	4
PbO <sub>2</sub> (M)	10
Pb Stearate	0.15

PART B

LP-2	20
LP-32	80
Santicizer 160	11
Titanox RA-50	7
T-606	0.25
Thixcin GR	8
Methyl Ethyl Ketone	10
Toluene	10

FORMULATION B:

<u>Base</u>	<u>Pbw</u>
LP-32	100
Cameltex	50
Multifex MM	20
Titanox RA-50	10
Silane A-187	0.7
Thixcin GR	8
Monoethanolamine	0.1
Methyl Ethyl Ketone	40
Xylene	40

Accelerator

Zinc Peroxide	10
Amax	2
HB-40	10

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Table 2 (Continued)

FORMULATION C:

Thiokol FA	100
Zinc Oxide	10
Sterling S	60
Stearic Acid	0.5
MBTS	0.3
DPG	0.1

Cure: 40 min/298°F

FORMULATION D:

Thiokol ST	100
Lime	1.
Zinc Peroxide	5.
Stearic Acid	1.
Sterling S	50.
Lithopone	50.

Cure: 30 min/310°F



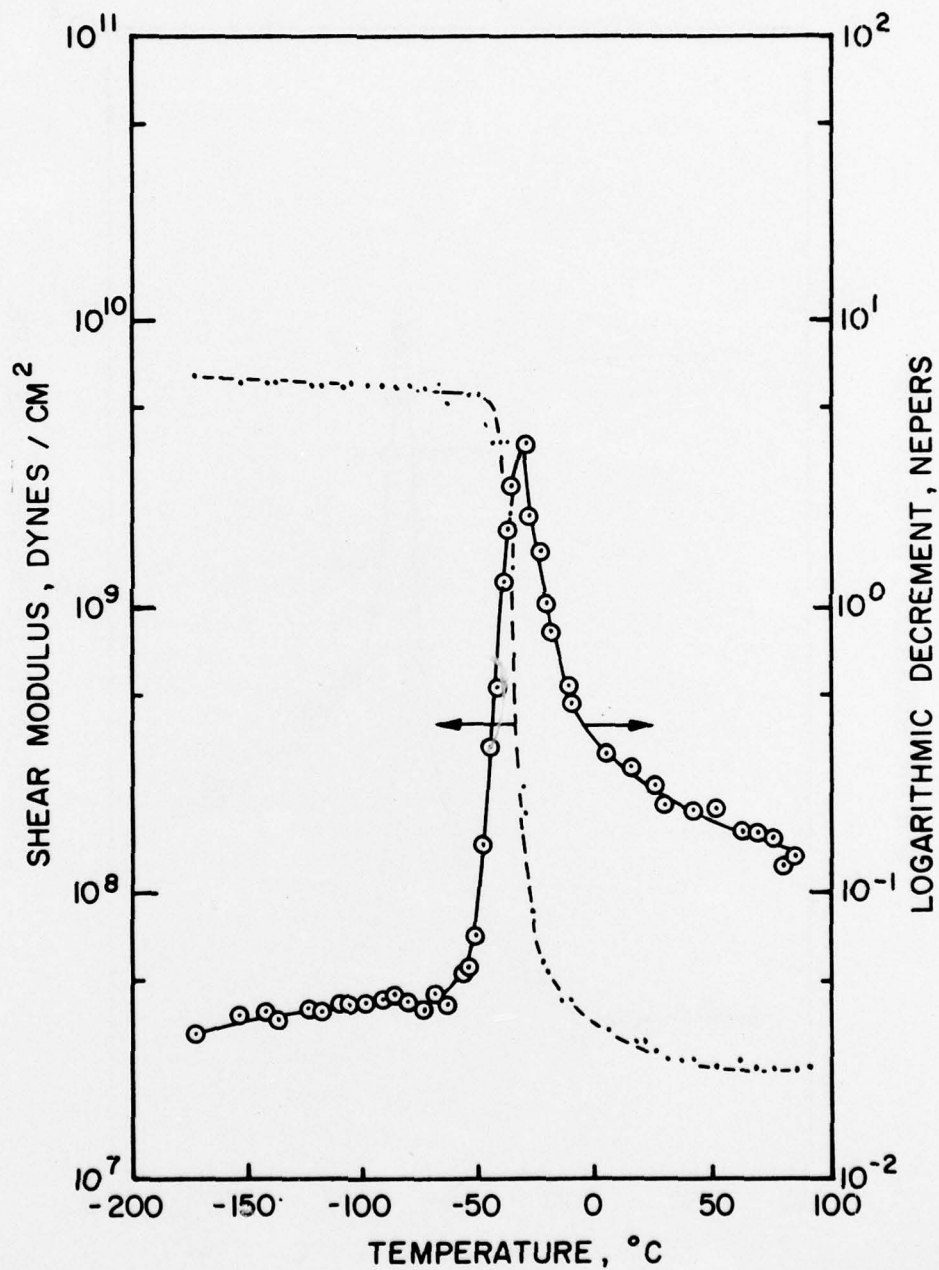


FIG. 1 SHEAR MODULUS & LOGARITHMIC DECREMENT VERSUS TEMPERATURE FOR 1,2-POLYBUTADIENE

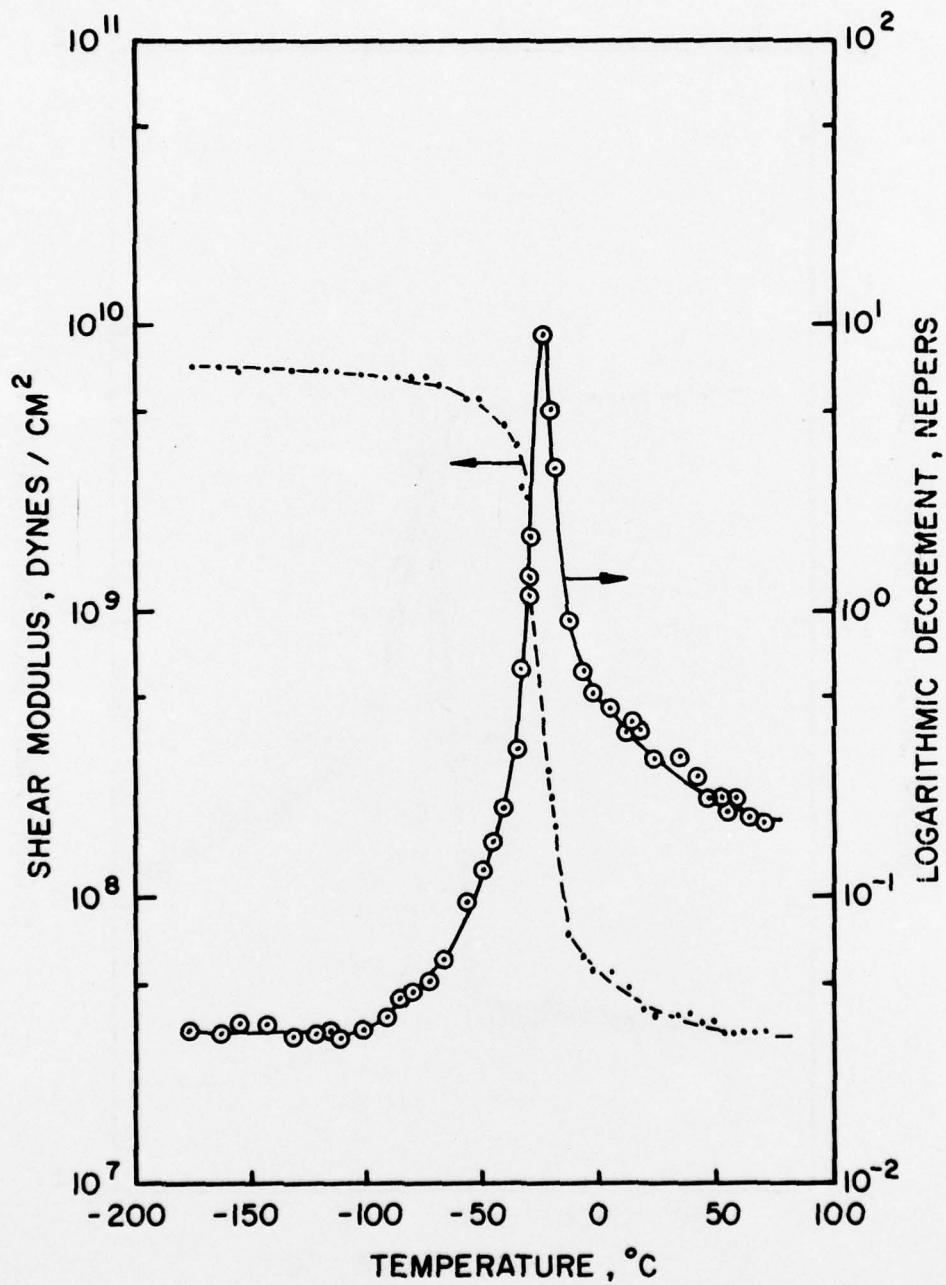


FIG.-2 SHEAR MODULUS & LOGARITHMIC DECREMENT VERSUS TEMPERATURE FOR NITRILE

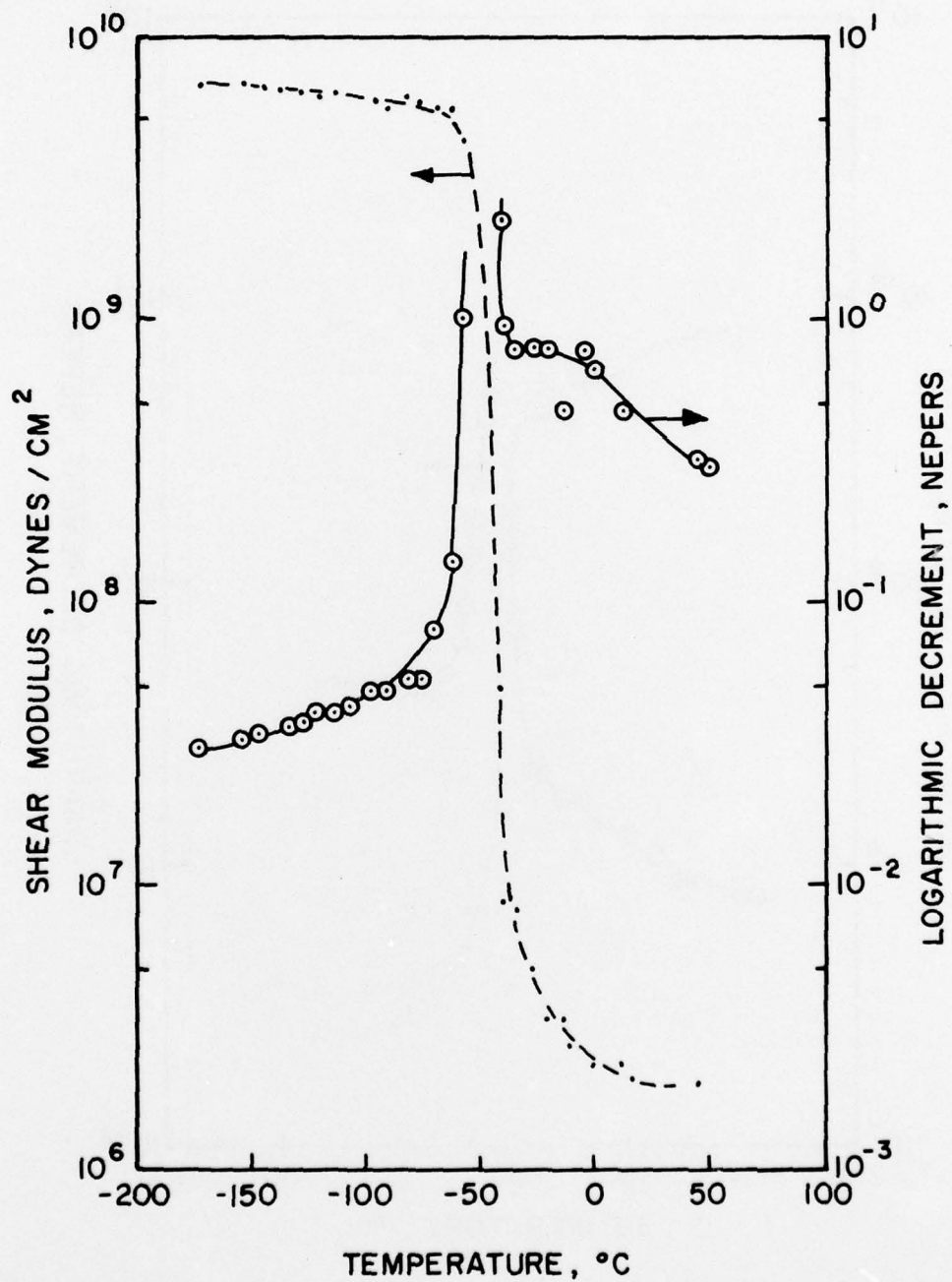


FIG.-3 SHEAR MODULUS & LOGARITHMIC DECREMENT VERSUS TEMPERATURE FOR POLYSULFIDE FORMULATION A

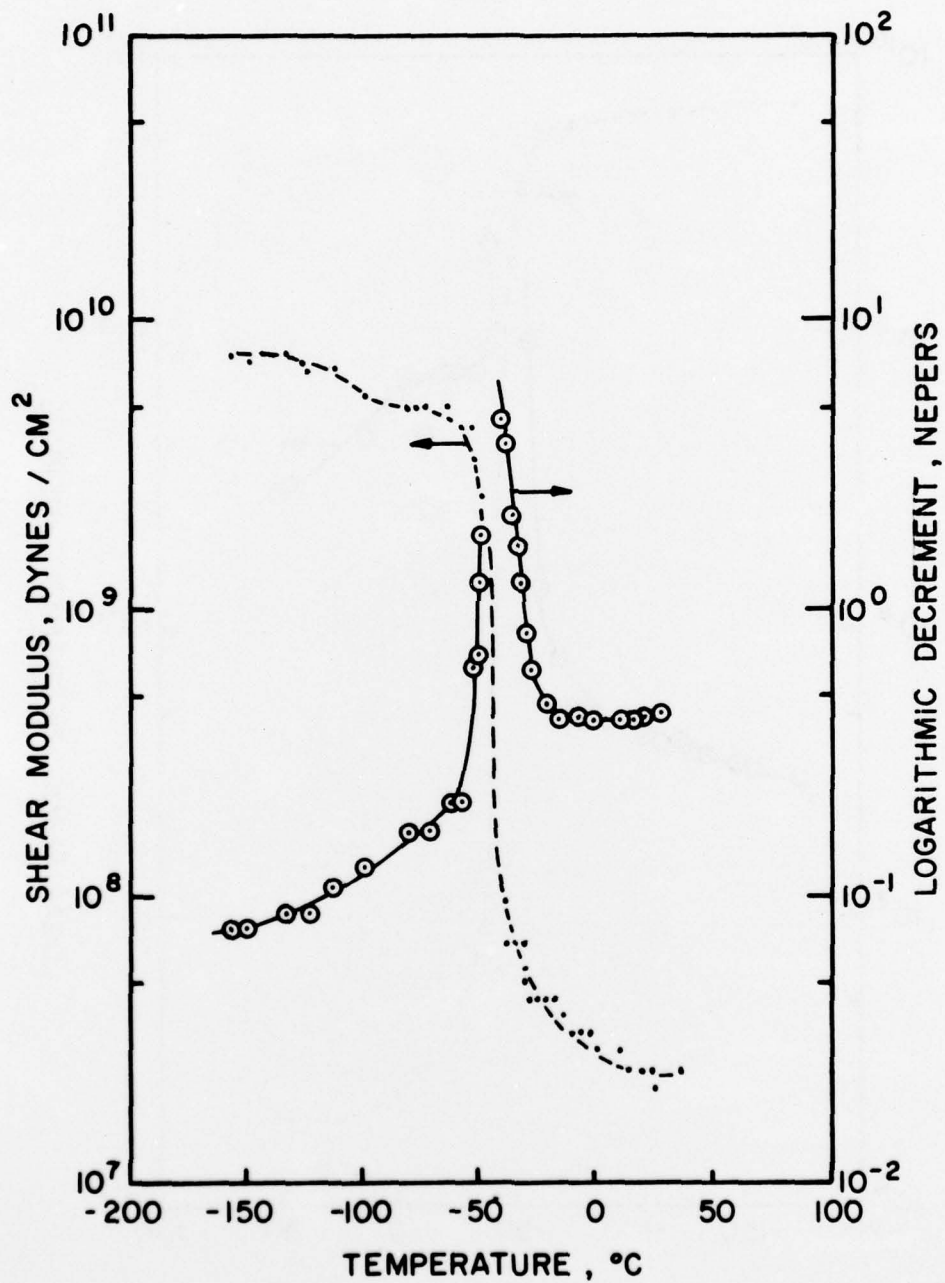


FIG. - 4 SHEAR MODULUS & LOGARITHMIC DECREMENT VERSUS TEMPERATURE FOR POLYSULFIDE FORMULATION B



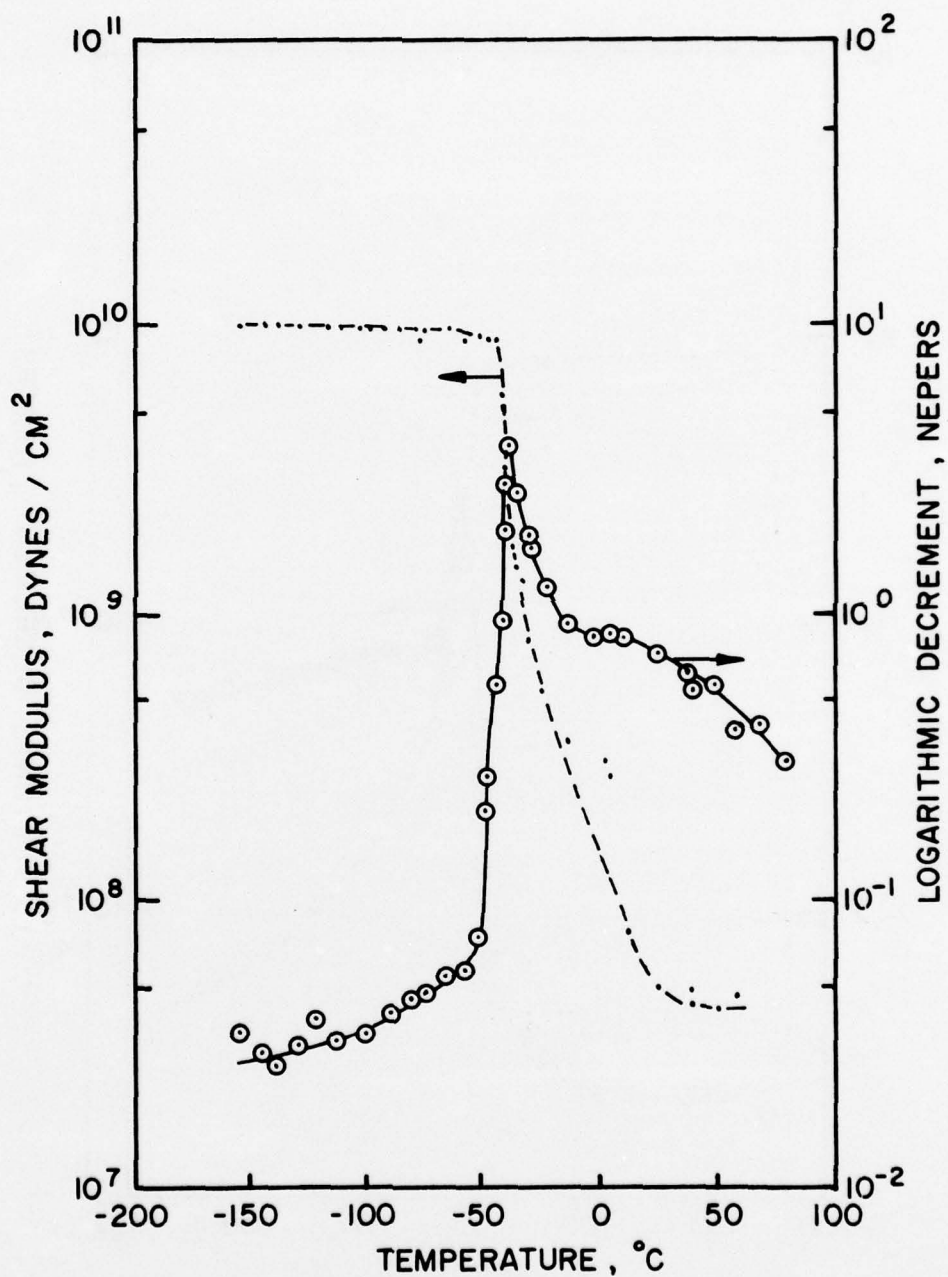


FIG.-5 SHEAR MODULUS & LOGARITHMIC DECREMENT VERSUS TEMPERATURE FOR POLYSULFIDE FORMULATION C

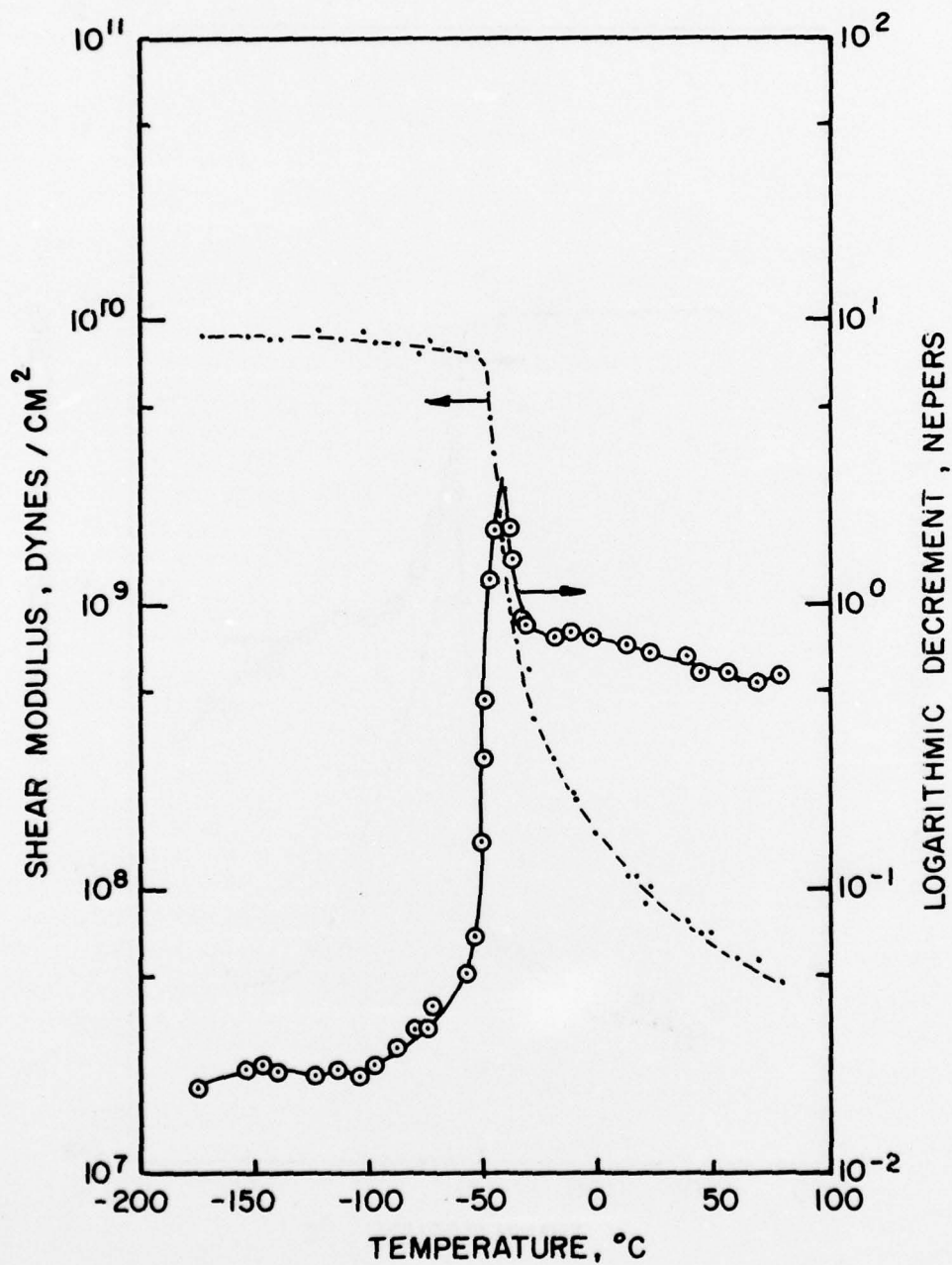


FIG.-6 SHEAR MODULUS & LOGARITHMIC DECREMENT VERSUS TEMPERATURE FOR POLYSULFIDE FORMULATION D

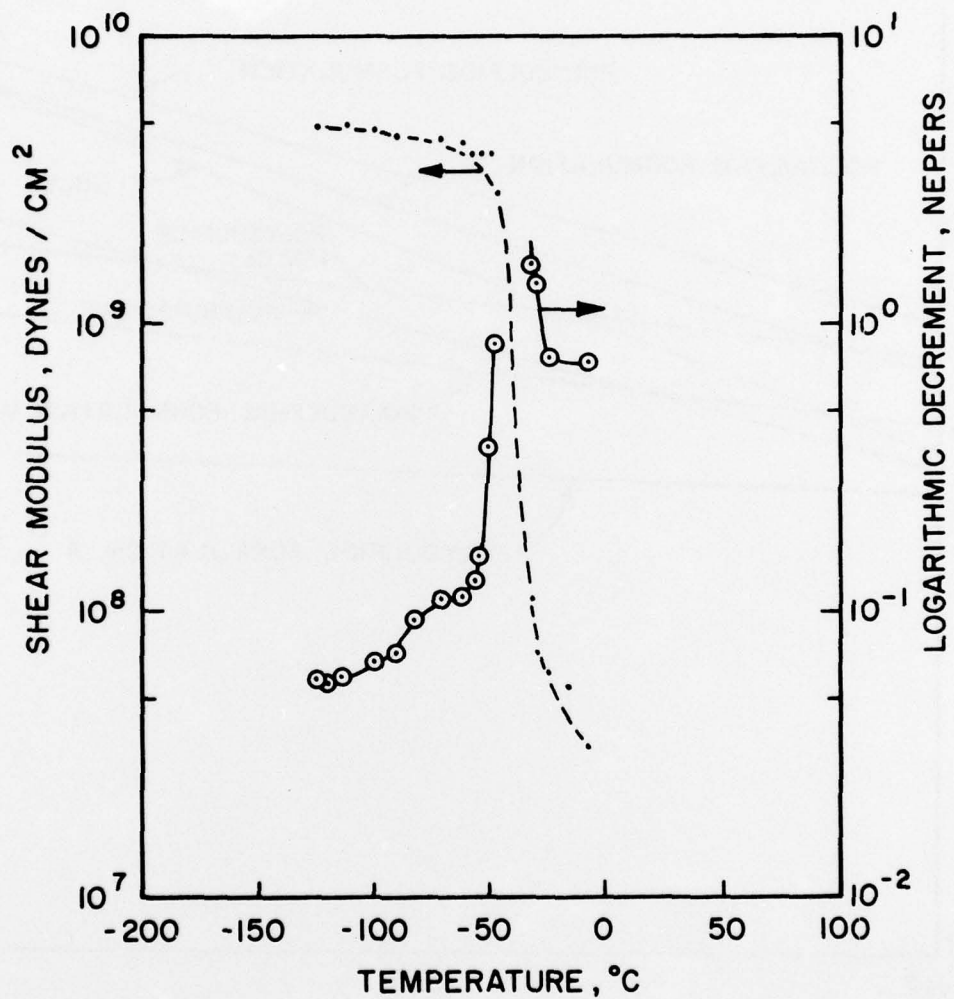


FIG. -7 SHEAR MODULUS & LOGARITHMIC DECREMENT VERSUS TEMPERATURE FOR POLYSULFIDE (3M Co.)



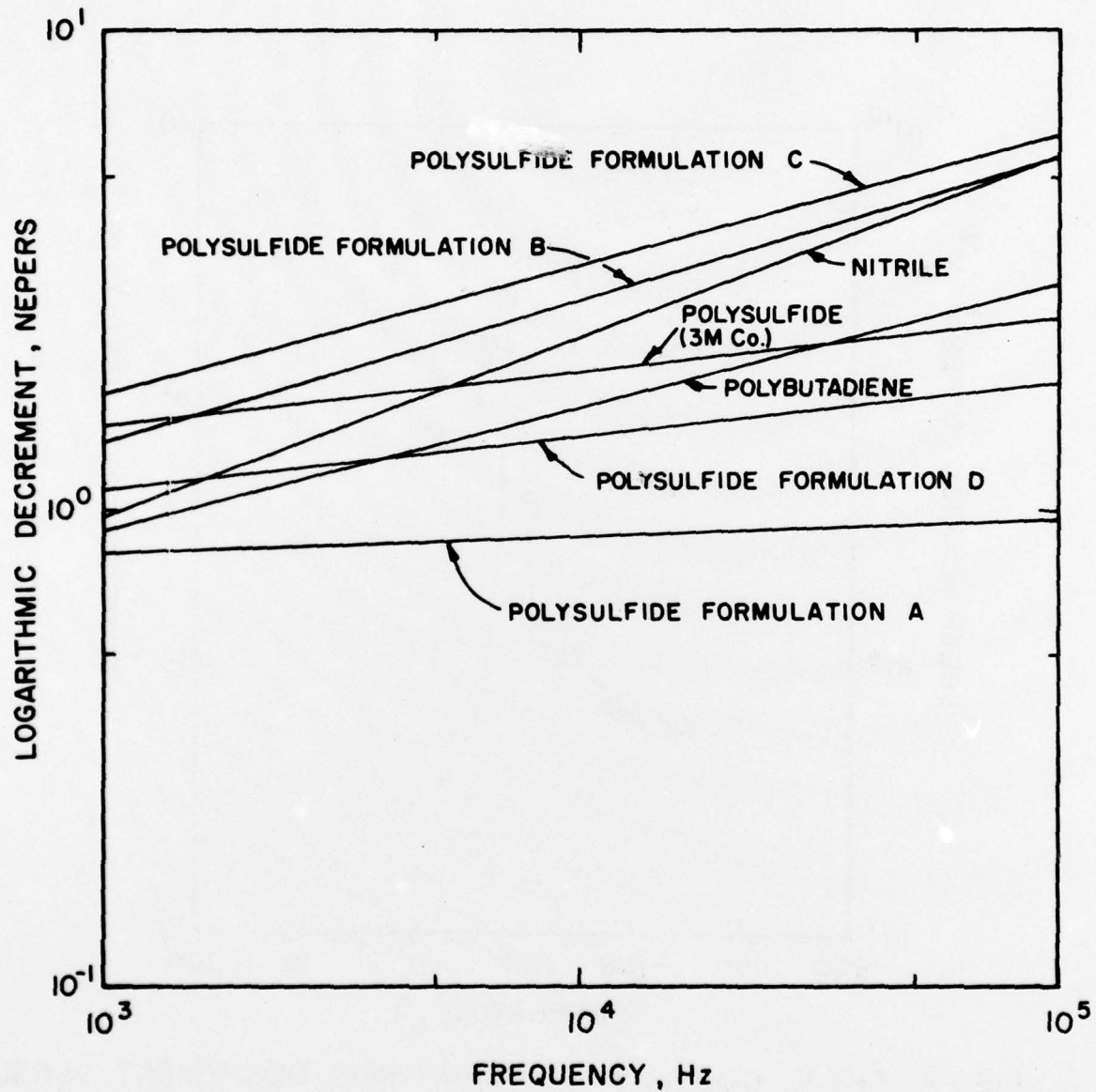


FIG. - 8 LOGARITHMIC DECREMENT VERSUS FREQUENCY  
FOR VARIOUS RUBBERS

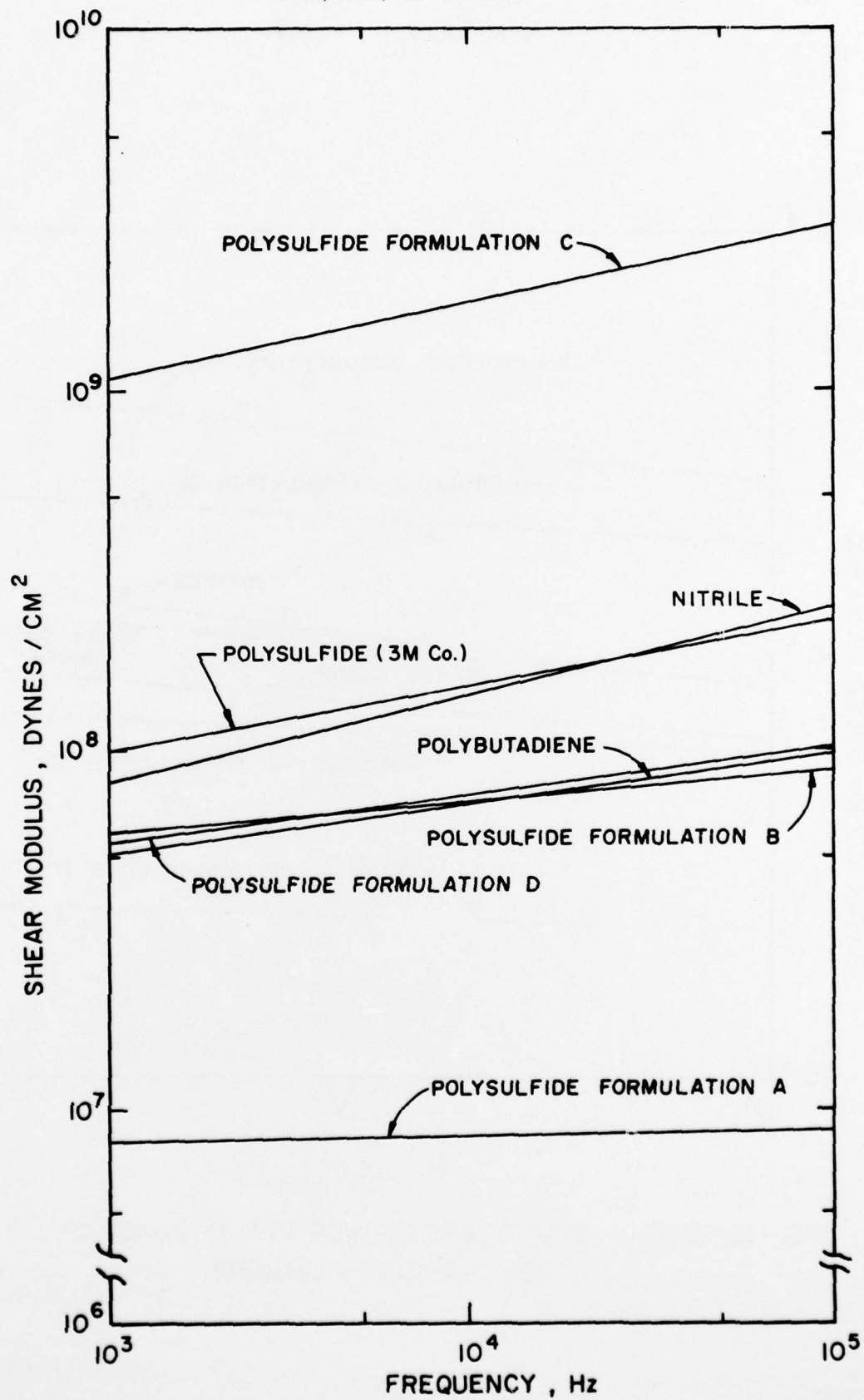


FIG.- 9 SHEAR MODULUS VERSUS FREQUENCY  
FOR VARIOUS RUBBERS

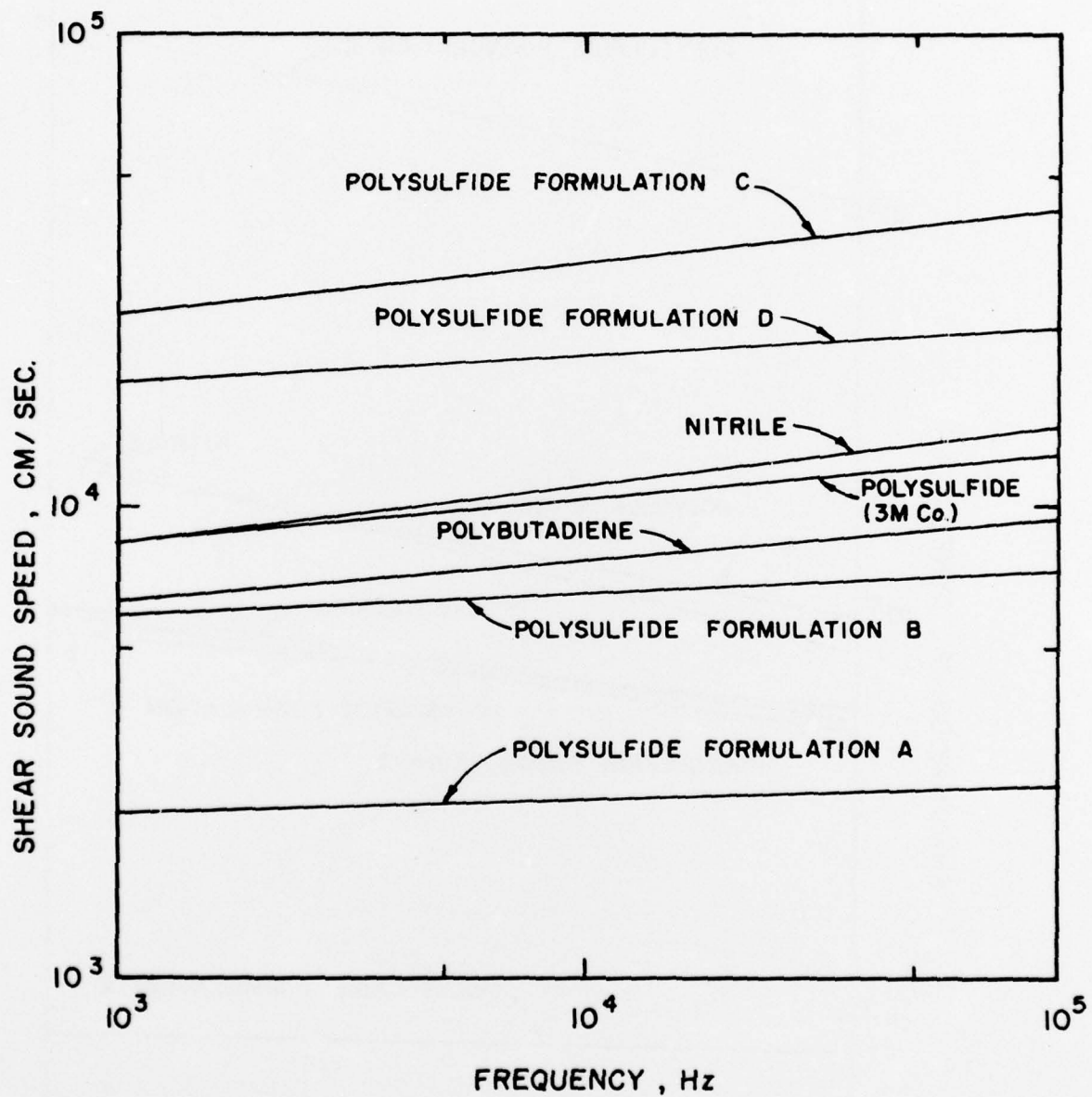


FIG.-10 SHEAR SOUND SPEED VERSUS FREQUENCY  
FOR VARIOUS RUBBERS

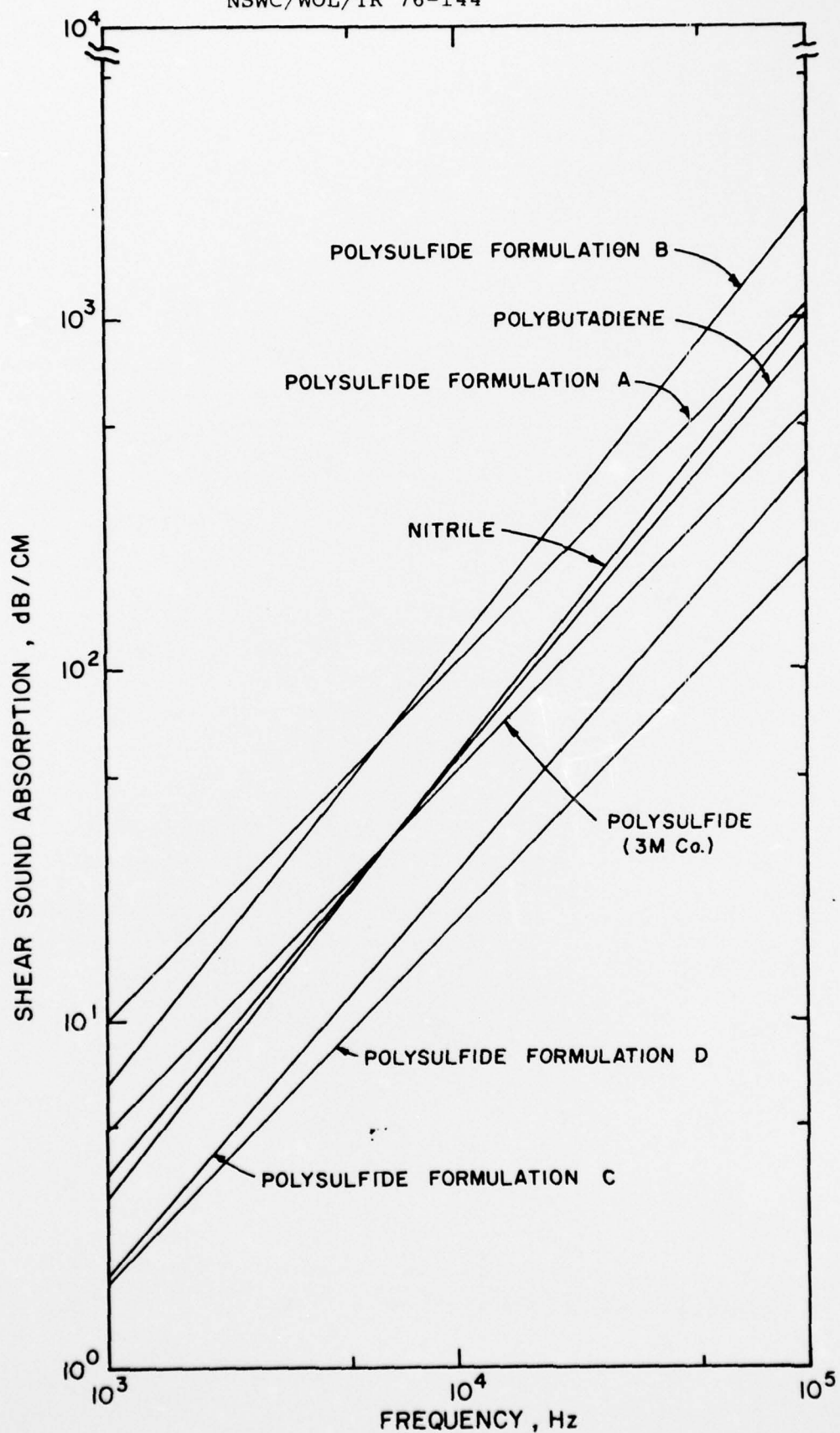


FIG. -II SHEAR SOUND ABSORPTION VERSUS FREQUENCY FOR VARIOUS RUBBERS



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